REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*: 11/4/08

2. REPORT TYPE: Final

3. DATES COVERED *(From - To)*: 6/15/07 - 6/14/08

4. TITLE AND SUBTITLE: Instrumentation for Nano-porous, Nano-particulate Geopolymeric Materials Research

5a. CONTRACT NUMBER: FA9550-07-1-0490

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S): Kriven, Waltraud M.

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AF Office of Scientific Research
875 N. Randolph St., Rm 3112
Arlington, VA 22203

10. SPONSOR/MONITOR'S ACRONYM(S): AFOSR

11. SPONSOR/MONITOR'S REPORT NUMBER(S): AFRL-OSR-VA-TR-2013-0962

12. DISTRIBUTION / AVAILABILITY STATEMENT
Unrestricted Availability

20130918296

13. SUPPLEMENTARY NOTES

14. ABSTRACT – Geopolymers are a new class of ceramic materials which are best understood as rigid inorganic, aluminosilicate, hydrated gels, charge-balanced by cations such as Na^{+1}, K^{+1}, or Cs^{+1}. The ease and versatility of forming, together with the tailorability of their properties (e.g. nano-porosity) qualify geopolymers as promising candidates for a variety of applications such as filters, catalyst supports, light weight, thermal shock resistant refractory composites, thermal/environmental barrier coatings, as well as smart composites. The PIs group, supported by AFOSR (Grant # FA 9550-06-1-0221) is working on tailoring the nano- and meso-porosity, and the microstructure of geopolymers and their composites. This grant was used to procure equipment to synthesize and characterize the nano- and meso-porous geopolymers, and study their evolution upon curing and conversion to ceramic phase. The instruments purchased included (a) Micromeritics ASAP 2020 equipped with chemisorption option and mass spectrometer, (b) TestEquity 1007H Temperature/Humidity Chamber, is deemed essential, (c) Union Process Model 01HD Attritor mill, and (d) Thinky Mixer ARE 250.

15. SUBJECT TERMS
Geopolymers; nano-porosity; microstructure; nano-particles; surface area

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
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FINAL REPORT

Project Title: Instrumentation for Nano-porous, Nano-particulate Geopolymeric Materials Research

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Contract Number: FA9550-07-1-0490

The primary objective of instrumentation purchased through this AFOSR-DURIP award (Grant # FA9550-07-1-0490) was to enable quantitative characterization of nano-porosity and mesoporosity in the microstructure of geopolymers, and their evolution upon curing and conversion to ceramic. The ability to understand and tailor the nano and meso-porosity and microstructure of geopolymers and their composites is part of an ongoing research project in the PIs research group, which has been supported by the AFOSR (Grant# FA 9550-06-1-0221). For this purpose, instrumentation for both characterization and processing of geopolymers was requested and included (a) Micromeritics ASAP 2020 Accelerated Surface Area and Porosimetry analyzer system equipped with a chemisorption option and mass spectrometer, (b) TestEquity 1007H Temperature/Humidity Chamber, is deemed essential, (c) Union Process Model 01HD Attritor mill, and (d) Thinky Mixer ARE 250.

All the above instruments were purchased through this grant, and are being used (or installed) to study nano-porous and nano-particulate geopolymeric materials.

The Micromeritics ASAP 2020 instrument was purchased and is being installed. It will enable quantitative evaluation of both the physical characteristics and physiochemical changes as a function of composition and processing conditions. The TestEquity 1007H Temperature/ Humidity Chamber, is being used to study the effect of humidity and temperature, the two most crucial factors that influence curing of geopolymers, on the geopolymer microstructure under a range of controlled conditions. Further results from these studies will be reported in the technical report for the respective AFOSR sponsored project.

The use of controlled humidity to properly cure, dry, and tailor geopolymer porosity has never been previously explored. As geopolymers are being increasingly considered for a number of high tech applications such as filters, precursors to glass ceramics, catalysts, and coatings, such a study stands to benefit to a number of areas. Previous work in the PIs research group had shown that although most geopolymers set in less than four hours at room temperature, they continue to react for months. Microstructurally, this involves coarsening of the small precipitates commonly observed via SEM. The use of controlled humidity above 50-60°C is expected to accelerate curing and other reaction processes in a more reasonable timeframe suitable for conclusive studies. This will further the understanding of the relationship between intrinsic microstructure, porosity, curing conditions and resultant properties.

The attritor mill and the Thinky ARE 250 mixer were purchased to improve the synthesis and processing of geopolymers and geopolymer composites. The attritor mill enables synthesis of nano-sized high surface area ceramic powders in geopolymers and geopolymeric composites. On the other hand, the Thinky ARE-250 mixer was purchased to assist with homogeneous dispersion of secondary phases in geopolymer composites. The THINKY ARE-250 Mixer is a non-contact "planetary" mixer which mixes, disperses, and degasses materials in seconds to minutes, in a sealed or lid-less container such as a jar, beaker, syringe tube, or cartridge. It is also effective in controlling shear, so as not to damage the materials. This product was evaluated specifically for use in geopolymer processing and our findings suggest that Thinky ARE-250 mixer provides exceptional advantages in mixing secondary phases, such as fibers and other nano-sized particulates. The mixing action (essential for reaction with metakaolin) and the ability to de-air (for removal of macropores) are also superior to an overhead mixer system which was used in the research project earlier.

The equipment purchased through this award is essential for the success of ongoing research in geopolymers by the PI (AFOSR Grant # FA 9550-06-1-0221). It is anticipated to catalyze the advancement of knowledge in this class of material system which holds strong promise in applications such as light weight, thermal shock resistant ceramic composites, coatings as well as smart composites.

Broader Impact

Geopolymers are a new class of ceramic materials which are best understood as rigid inorganic, aluminosilicate, hydrated gels, charge-balanced by cations such as Na^{+1}, K^{+1}, or Cs^{+1}. The microstructure of geopolymers is nano-porous (~ 3.4 nm radius), nano-particulate ($\leq$ 5 nm in size), and the porosity constitutes over 40% of the body by volume. The ease and versatility of forming, together with the tailorability of their properties (e.g. nano-porosity) qualify geopolymers as promising candidates for a variety of applications such as filters, catalyst supports, light weight, thermal shock resistant refractory composites, thermal/environmental barrier coatings, hydrogen storage materials, as well as smart composite materials (e.g. sensors). In order to realize the full potential of this material, it is imperative to develop a fundamental understanding of the geopolymer microstructure, leading to its evolution into a polycrystalline ceramic upon heating. The lack of quantitative material science knowledge is an impediment to the advancement of this class of material. The current stage of geopolymer research is fraught at the elementary stage of porosity engineering and control. Success at this stage has the potential to usher in a variety of high tech applications for this very promising material.

Instrumentation purchased through this grant was aimed to overcome the equipment limitation being faced in the awarded research contract (AFOSR Grant # FA 9550-06-1-0221), and thus facilitate superior quality research deliverables also. This instrumentation complements the equipment available to the PI for research at UIUC. It will certainly transform the way research is conducted on geopolymer materials.